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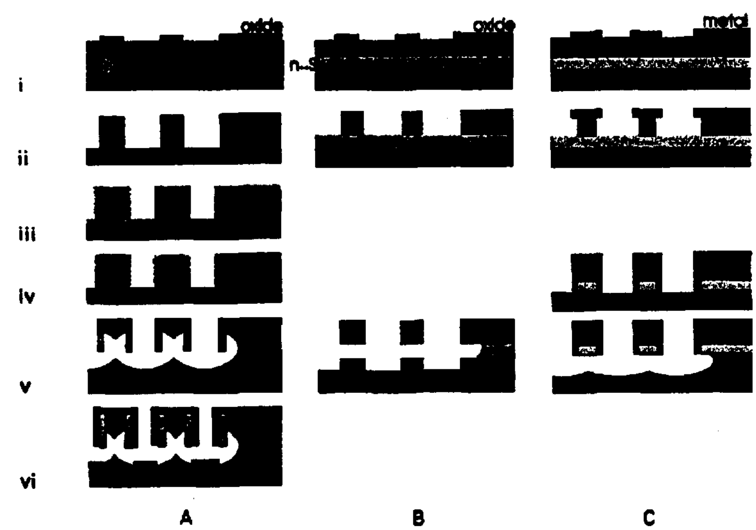

(57) Abstract

A process for producing etched micromechanical structures is provided, using Reactive Ion Etching (RIE), wherein a substrate is etched with a silicon etch gas mixture to obtain an aspect ratio of at least 10. The process comprises the steps of: a) anisotropic etching using a first silicon etch gas to obtain a primary microstructure; b) depositing a halocarbon film on the walls of the primary microstructure; d) isotropic etching using a second silicon etch gas, to obtain a final microstructure; said steps being carried out in a single run. Optional further steps are: c) etching the floor of the primary microstructure using said first silicon etch gas; and e) depositing a halocarbon film on the surface of the final microstructure. The process may involve applying high pressure (5-30 Pa) and low energy (10-90 eV), and preferably the use of a sulphur hexafluoride/oxygen/trifluoromethane plasma. The process can be controlled by monitoring the blackening of a silicon test surface as a function of varying the process parameters.

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PROCESS FOR PRODUCING MICROMECHANICAL STRUCTURES BY MEANS OF REACTIVE ION ETCHING

FIELD OF THE INVENTION

The invention relates to a process for producing micro(electro)mechanical structures in a substrate using standard Reactive Ion Etching (RIE), wherein the substrate is etched with a silicon etch gas mixture. The invention relates in particular to such a RIE process that can be carried out in a single run.

INTRODUCTION

Profile control is important in microtechnology. Especially vertical walls are needed in order to obtain high feature densities. Most commonly, wet chemical etchants are used to create anisotropic profiles in silicon, because they are inexpensive and easy to use. However, the etched profile is controlled by the crystal orientation, so there is minor freedom in etching different tapered profiles. Dry plasma etching is becoming a standard tool in microtechnology. Although the basic investments are much higher in dry etching, it is possible to etch controllable profiles without using the crystal orientation or doping. Plasma etching can be divided into three main groups; the physical ion beam etching (IBE), the synergetic reactive ion etching (RIE), and the chemical plasma etching (PE). Generally, IBE shows only positively tapered profiles, low etch rates, and low selectivity, whereas PE gives rise to isotropic profiles, high etch rates, and high selectivity. In RIE it is possible to provide the plasma with a chemical etchant for the etching of the substrate, a passivator for blocking the etching at the sidewalls of a trench, and an ion source for the local removal of the passivation layer at the bottom of the etching trenches. When these processes are controlled in the correct manner, it is possible to create all kinds of trenches with excellent profile control, high etch rates and selectivity. RIE processes which use the deposition of a passivating film are called: ion-inhibitor RIE. When etching polymers, it is not necessary to passivate the trench sidewalls. In these cases the etching is possible because of ion bombardment. This is a typical reactive ion beam etching (RIBE) process, but can also be fulfilled at higher pressures with an RIE apparatus. RIE processes which use only the incoming ions are called; ion-induced RIE. To increase the etch rate, standard RIE is modified to create a higher density plasma [1-6], but these etchers are expensive and therefore less attractive.

Normally, halogen-based plasmas are used for the chemical etching of silicon, because of their high etch rates [1-13]. Except for the fluorine-based plasmas, these gases are particularly hazardous (e.g. chlorine, bromine, and chlorinated compounds) and special precautions are recommended.

The passivation layer can be grown: **1** from polymer precursors which are lead into the plasma [8-10], **2** by resputtering mask material [11], **3** by inserting gases which act as an oxidant (forming siliconoxyhalogen) [1,12,13], or **4** by freezing the normally volatile reaction products of the silicon with the radicals at the trench walls [3,4]. The deposition of a halocarbon polymer film has the disadvantage that this film is thermally less stable than a growing inorganic siliconoxyhalogen film and the freezing of reaction products uses the expensive (cryogenic) coolers. The resputtering of mask material is not acceptable because areas which should stay clean are also contaminated. Because the passivating film is very thin the incoming ions should not be highly energetic, so the selectivity will be very high and the substrate damage will be low. Also, because of the low energy of the ions, trenching and faceting are not found and it is very easy to change the direction of the impinging ions thus changing the etched profile. A major problem during etching silicon vertically is the forming of "grass" on the silicon surface, because of all kinds of micromasks deposited or grown on the silicon.

Commonly, low pressure oxygen plasmas are used for etching polymers. However, such a plasma creates a high d.c. self-bias voltage which is responsible for substrate- and mask-damage. During etching a polymer in low pressure RIE identical grass problems are observed as in the case of etching silicon.

Etching rates and profiles are observed to depend on feature size (i.e. aspect ratio dependent etching: ARDE) and pattern density (i.e. microloading) for Si, SiO_2, polymers, metals and group III-V elements also referred to as microscopic non-uniformities [15].

After etching micromechanical silicon structures, they often have to be released. This is not straightforward and many techniques have been proposed. Frequently, in surface micromachining, an intermediate SiO_2 layer of a silicon-on-insulator (SOI) wafer is used as a sacrificial layer which is etched using wet or vapour etchants. They are inexpensive but suffer from the so-called sticking problem, due to surface tension of liquids, and many solutions have been proposed to solve this problem [16]. In bulk micromachining some very useful dry plasma release techniques have been proposed [9,17]. Sticking is not found in dry etching, thus making this technique more reliable.

The aim of this work is the production of high aspect ratio (depth/width) features for use in Micro Electro Mechanical Systems (MEMS), without the specific constraints that are always attached to the previously proposed release techniques - wet, vapour, and dry.

DESCRIPTION OF THE INVENTION

The main object of the invention is to provide a process for manufacturing micro-(electro)mechanical systems (MEMS) in a single run by means of Reactive Ion Etching (RIE) using a halogen-based, especially a fluorine-based plasma and different types of substrate materials.

To achieve this it is another object of the present invention to provide deep trenches in to a substrate by means of reactive ion etching (RIE) using a fluorine-based plasma, not depending on crystal orientation and doping.

A third object of the present invention is to achieve highly controllable trench profiles by means of standard RIE.

It is another object of the invention to provide deep trenches with high aspect ratio, especially of 10 or more.

A next object of the present invention is to achieve high etch rates with good uniformity over the wafer.

A further object of the invention is to use high pressure RIE (> 50 mTorr) during the trench etching in order to obtain very low d.c. self-bias voltages (down to 10 V) in order to prevent electronics already in the substrate to be damaged. Due to the low bias voltage it is easy to bend the incoming ions to the sidewalls in order to create all kinds of profiles. The ion energy is between 10 en 90 eV, preferably between 10 and 50, particularly between 10 and 20 eV. Such low voltages are unique for relative simple RIE equipments. As a further important advantage of the low ion energy, the very high mask selectivity is mentioned. So only very thin metal layers (< 50 nm) will suffice to etch deep trenches.

A still further object of the invention is to add CHF_3 or another halocarbon in to the SF_6/O_2 plasma to prevent the forming of grass to get very smooth sidewalls and bottoms. At the same time it is possible to control the trench profile very easy and accurate with this halocarbon adding.

Another object of the present invention is to use the Black Silicon Method together with the profiles and d.c. bias voltage diagrams. The diagrams include the influence of the oxygen and CHF_3 content and the influence of the power and pressure on the trench profile and d.c. bias voltage. Herein, the formation of "grass" is used to find the desired profile.

Yet another object is to prevent ARDE effects such as RIE lag and sidewall bowing by varying the gas mixture while using the BSM.

As a further object of the invention, it is possible to provide extremely sharp tips for e.g. scanning tunnelling microscopy (STM). The tip radius is smaller than 5 nm while using an insulating mask for the tip mask [17].

The one-run multi-step process proposed according to the invention is a more sophisticated dry release technique able to extend the limits of microtechnology. As an example, a MEM comb-driven xy-stage is given.

According to the invention, very deep trenches (up to 200 μm) with high aspect ratios (of about 10 or higher) in silicon and polymers can be etched using a fluorine-based plasma, such as $SF_6/O_2/CHF_3$. Isotropic, positively and negatively (i.e. reverse) tapered as well as fully vertical walls with smooth surfaces are achieved in silicon or in polymers by controlling the plasma chemistry, which is independent of crystal orientation and doping. A convenient way to find the processing conditions needed for a vertical wall is described: the Black Silicon Method. This new procedure is checked for three different Reactive Ion Etchers (RIE); two parallel plate reactors and a hexode. The influence of the r.f. power, pressure, and gas composition on the profile is shown. Micro Electro Mechanical Systems (MEMS) can be manufactured in a on-run multi-step dry RIE process which uses e.g. commercially available silicon on insulator (SOI) wafers.

These objects are achieved by a process for producing micromechanical structures using Reactive Ion Etching (RIE), wherein a substrate is etched with a silicon etch gas mixture, comprising the steps of:

a) anisotropic etching using a first silicon etch gas to obtain a primary microstructure;

b) depositing a halocarbon film on the walls of the primary microstructure;

d) isotropic etching using a second silicon etch gas, to obtain a final microstructure;

said steps being carried out in a single run.

Preferably, the process comprises, after step b), the step of:

c) etching the floor of the primary microstructure using said first silicon etch gas.

As an advantageous optional final step, the process according to the invention further comprises the step of:

e) depositing a halocarbon film on the surface of the final microstructure.

THE SYNERGETIC MECHANISM OF $SF_6/O_2/CHF_3$ PLASMAS

In an $SF_6/O_2/CHF_3$ plasma, each gas has a specific function and influence, so the

etched profile is easily controlled just by changing the flow rate of one of these gases. In such a plasma SF_6 produces the F* radicals for the chemical etching of the silicon forming the volatile SiF_4, O_2 creates the O* radicals to passivate the silicon surface with SiO_xF_y, and CHF_3 (or another halocarbon) is the source for the CF_x+ (or other halocarbon) ions which etch the SiO_xF_y layer in one direction forming the volatile CO_xF_y. Of course, SF_x+ ions are also able to remove the oxyfluoride by way of the volatile SO_xF_y gases, but the SF_6 flow is fixed on the O_2 flow to ensure a vertical wall. Thus, the CHF_3 gas is a nearly independent source of oxyfluoride etching ions.

A more or less contrary mechanism can also explain the directional etching. In this mechanism the CF_x* species are passivating the silicon surface which are etched by way of imparting O+ ions. However, this mechanism is less likely in the pressure regime used in our study as will be clarified further on.

The $SF_6/O_2/CHF_3$ chemistry allows etching of highly controllable profiles in silicon at very low ion energies (10–90eV) and high etch rates (up to 5 μm/min). The low ion energy prevents substrate damage (electronics), mask erosion (the selectivity to metal masks is practically infinite), and makes it easy to change the profile of the trench. The ion energy is ruled by the potential which is developed between the plasma and the powered electrode; the d.c. self–bias. The d.c. self–bias decreases when the power decreases or the pressure increases. The pressure may be e.g. from 25 to 250 mTorr (3.3 – 33.3 Pa), in particular from 50 to 200 mTorr (6.7 – 26.7 Pa). Gases like O_2 and CHF_3 create high bias voltages whereas SF_6 gives rise to a very low voltage. Thus, when the oxygen flow is increased the d.c. self–bias also increases and ions will gain more energy before colliding with the substrate surface.

In etching silicon with the $SF_6/O_2/CHF_3$ mixture there is a constant competition between the etching fluorine radicals and the passivating oxygen radicals. The etching is increased directionally by way of the CF_x+ ions. When the SF_6 content is increased, the formation of the blocking layer is less pronounced and therefore the profile will be more isotropic (i.e. PE–like). Increasing the oxygen content will decrease the chemical etching and the etch mechanism will become less isotropic. At higher oxygen concentration the etching will become physical which results in positively tapered profiles (i.e. IBE–like). Increasing the CHF_3 content will increase the removal of the blocking layer, thus making the profile less positively tapered. Moreover, the ions are charged positively, whereas the substrate is negatively biased and because of this mechanism, it is possible to create negatively (i.e.

reverse) tapered profiles due to ion bowing. At higher CHF_3 concentration CF_x specimens will scavenge the oxygen radicals, thus preventing the blocking layer to form, which results in a more isotropic profile. When the power, pressure, and flows are in the correct balance, vertical wall profiles result. Two less attractive effects may be observed here: *1* When the line spacing is wider, the trenches are deeper. This effect is known as "RIE-lag". *2* The bigger areas have negatively tapered wall profiles because of ion bowing. These ARDE effects can be reduced by making the silicon trench walls more insulating-like and decreasing the d.c. bias voltage, thus increasing the passivator. To ensure a certain profile the ion flux has to be increased at the same time conform the BSM. A higher pressure or lower power results in a more positively tapered profile, because the energy of the impinging ions is lower (d.c. self-bias). In these cases, of-normal ions are more likely to reflect from the sidewalls without etching it. When the etching is performed in the isotropic or negatively tapered regime, thus at low oxygen, high CHF_3 flow, low pressure, or high r.f. power, micromasks such as native oxide, dust, or resputtered mask material will be constantly underetched and etched surfaces are staying smooth preventing the forming of grass.

It is also possible to etch polymers, instead of silicon, with high etch rates and aspect ratios with the same $SF_6/O_2/CHF_3$ plasma, but in these cases the specific function of each gas is different. Now, O_2 creates the O* radicals to etch the polymer chemically, but this etching is highly temperature dependent. At temperatures near or above the polymer glass transition temperature, the etch profile is purely isotropic and the etch rate can be as high as 5 microns per minute. This process is normally fulfilled in so-called plasma ashing systems to strip resist after mask duplication. In order to create anisotropic profiles, it is necessary to block this thermo-chemical etching, so the substrate is cooled at room temperature (20 °C) and at the same time O+ ions are used to etch the trench bottom. For this reason RIE is used to direct the ions from the plasma glow region towards the substrate. Because of the highly anisotropic etch, grass will appear just as in the case of the RIE of silicon. To prevent this formation of grass, again CHF_3 is added resulting in nearly vertical and smooth surfaces. To lower the high d.c. bias voltage (thus increasing the mask selectivity) which is created during O_2/CHF_3 reactive ion etching SF_6 and silicon is added giving very low bias voltages (down to 10 eV). The addition of CHF_3 as well as SF_6 is not influencing the polymer etch rate (up to 2 μm/min) more than 10%.

Although the $SF_6/O_2/CHF_3$ plasma is described here as suitable for the process of

the invention, the process also works with other silicon etch gases e.g. CF_4, NF_3, SiF_4, CF_3Br, CCl_4 or Cl_2. In fact, every plasma mixture which consists of a chemical etchant, a passivator and an ion source can be used, even when the substrate is not silicon, but e.g. a polymer.

Instead of trifluoromethane (CHF_3), other halocarbons can be used as a source of $CHal_x^*$ species. Examples include other halomethanes and haloethanes containing a plurality of fluorine and/or chlorine atoms, preferably fluorine atoms. If the halocarbon does not contain hydrogen atoms (such as CF_4 and C_2F_6), it is preferred that hydrogen (H_2) is added.

When the present process is used in etching silicon structures, the silicon loading of the substrate should be sufficiently high, e.g. at least 10%. If the substrate has a lower silicon loading, additional silicon may be added to the etching chamber in any form.

The present process can also be used for etching polymer structures such as polyimides, polycarbonates, polyacrylates (PMMA) and polystyrene.

THE BLACK SILICON METHOD

A strong method is found to determine the vertical profile regime. This method uses the fact that the silicon is turned black when the vertical wall recipe is found. This method will be called the "Black Silicon Method". Before the Black Silicon Method is formulated below, the reason for this effect will be explained and a way to get rid of this blackening will be described.

The origin of black silicon: As stated, there is a constant competition between the fluorine radicals that etch and the oxygen radicals that passivate the silicon. At a certain oxygen content there is such a balance between the etching and the passivation that a nearly vertical wall results. At the same moment native oxide, dust, etcetera will act as micromasks and, because of the directional etching, spikes will appear. These spikes consist of a silicon body with a thin passivating siliconoxyfluoride skin. They will become higher in time and, depending on the etch rate, they will exceed the wavelength of incoming light after some time. This light will be "caught" in the areas between the spikes and cannot leave the silicon surface any more. So, all the light is collected by the etching surface and it is turned into black. In fact, this optical diffuser could be used for all kinds of applications where the reflection of light from the surrounding is not desired, e.g. laser applications or sunlight collectors. An etched silicon piece under directional conditions may result in spikes which are 50μm in height and a few μm in width. The origin of micromasks is caused by native oxide, dust, and so on, which is already on the wafer before etching. However, they are also

formed during the etching because silicon oxide particles coming from the plasma are adsorbing at the silicon surface or because of the oxidation of the silicon surface together with the angle dependent ion etching of this oxide layer. Another source of particles during etching which will act as micromasks is the resputtering of mask material due to imparting ions.

Preventing black silicon: Spikes which are formed because of dirty wafers before etching are easily controlled by giving the wafer a precleaning step. For instance, native oxide can be removed with the help of an HF dip and dust is less a problem when using the lift-off technique in applying the mask layer, instead of the normally used chemical etching of the mask material with the help of a resist pattern. However, the micromasks which originate during etching must be controlled in a different way. First of all, the resputtering of mask material can be suppressed when the ion energy is low or when the right materials are chosen. The silicon oxide particles are less a problem when the selectivity between the silicon and the silicon oxide is minimised, but this only occurs when the incoming ions are highly energetic and at these moments the process is not favourable any more because of substrate damage and the just mentioned mask erosion. As already stated, it is possible to prevent spikes from forming by constantly underetching the micromasks isotropically or etching the features with a slightly negative undercut. The isotropic solution makes only sense when it is used as a post etch, because otherwise the feature density is limited. On the other hand, the negative underetching is an excellent way to control the smoothness of the substrate surface barely limiting the feature size density. In this study the addition of CHF_3 to an SF_6/O_2 plasma is described and its ability to prevent grass. Yet another approach to attack the grass problem is the application of different masks.

The Black Silicon Method: In this section, an easy way to find the vertical wall regime is described with the help of the information already given. A more or less general tool is reached in which the recipe for any RIE system can be found just by fulfilling the sequence written down below. As can be concluded from point 3 of this sequence purely vertical walls can be achieved for any pressure, power, O_2, CHF_3, or SF_6 flow. This is an important conclusion because now we are able to create any d.c. self-bias we want without changing the profile. For instance, it is possible to develop very low bias voltages (< 20 eV) at the higher pressures giving very high mask selectivity, maintaining the profile. In such cases the etched silicon bottom and the sidewalls are perfect. It is also observed in the diagram of figure 3 that a vertical wall profile is found for zero CHF_3 flow. This means that

the passivation with siliconoxyfluorides at the sidewalls is more likely than the passivation with a fluorocarbon layer, although it is still possible that at different pressure, loading, etcetera, the fluorocarbon layer is more pronounced. Also, the observation that increasing the oxygen flow gives rise to a more positively tapered profile is a strong indication that siliconoxyfluoride is the sidewall passivator. Auger analysis showed that indeed the sidewalls are covered with silicon oxide; there is no carbonic species found.

The Black Silicon Method is tested for three different RIE systems. Most experiments are performed with a plan parallel plate reactor "plasmafab 340" from the STS company and a second plan parallel plate single wafer reactor "plasmatherm 500" showed identical results. A third system, the hexode "AME-8100" from Applied Materials, is used for the batch fabrication of silicon wafers and is also able to achieve vertical profiles. However, the etch rates are approximately one order in magnitude lower than for the single wafer etchers and for this reason less powerful. This is because the wafers are much longer exposed to the aggressive plasma chemistry giving rise to surface roughening when etching very deep trenches in silicon. The etch rate can be increased by decreasing the reactor loading. However, decreasing the exposed silicon surface area too much (typically less than 10% "open" silicon surface on the substrate wafer) will change the plasma chemistry (thus changing the profile) beyond a point where adjustment of the variables such as pressure, oxygen, flow, etc., can no longer prevent the underetching of the mask. Nevertheless, it is possible to insert some extra silicon in the reactor to ensure a minimal loading.

A formulation of the Black Silicon Method:

1. Place a piece of silicon in the reactor and adjust the preferred power and pressure for an SF_6/O_2 plasma. Etch ca. 1 micron of silicon, open the process chamber, and look if the silicon is black. If not, do the same again but increase the oxygen flow. Proceed with this sequence until the wafer is black. Increasing the oxygen too much, still will give rise to black, or better grey, silicon since there exists a positively tapered profile without any underetching. Alternatively, it is possible to sense the black silicon with the help of a laser/photodetector set-up.

2. After the black silicon regime is found add some CHF_3 to the mixture and increase this flow until the wafer is clean again. Too much CHF_3 will make the profiles isotropic (and smooth) because the CF_x species are scavenging the oxygen radicals which are needed for the blocking layer.

3. Now a wafer with the mask pattern of interest is inserted in the reactor and the

etched profile is checked. If necessary, add some silicon into the reactor chamber until the exposed silicon area is the same as in step 1 and 2. Increasing the SF_6 content will create an isotropic profile. Adding too much oxygen will make the profile positively tapered and extra CHF3 will make it more negatively tapered. Adding at the same time O_2 and CHF_3 with the correct balance will create very smooth and nearly vertical walls. Increasing the pressure or decreasing the power will make the profile more positively tapered. In figure 3 the influence of the O_2/CHF_3 flow and the pressure/power on the profile is given. Increasing at the same time the O_2 and CHF_3 flow or power, increasing the O_2 flow while decreasing the pressure, decreasing the pressure and power or CHF_3 flow and decreasing the CHF_3 flow while increasing the power, will hardly change the profile. However, such an change will increase the d.c. self-bias and a higher d.c. self bias will give the of-normal ions enough energy to etch the sidewalls, thus changing the profile a little. Structure heights of 100 micron with an undercut of less than 1 micron are achieved. In order to reduce ARDE effects, increase the CHF_3 and O_2 flow at the same time while maintaining the tapering of the trench.

Sharp positively tapered silicon tips for AFM applications can be fabricated with the BSM in allowing a controllable underetching. It is possible to fabricate spikes having an aspect ratio of 50 or more and a tip radius smaller than 5 nm. To achieve such sharp tips a remarkable phenomena is used which occurs during the RIE of these tips. When an insulating mask is used for the pattern transfer, this mask will slip over after the mask is completely underetched. This is caused by electrostatic forces which exist during the RIE of silicon. This mask is protecting the sharp tip after that moment from incoming energetic ions, so overetching is not a big problem.

The Black Silicon Method was developed for silicon trench etching, but it is found that this method works for polymer trench etching as well. Although the appearance of a polymer surface after anisotropic etching is not black but rather diffuse, the mechanism is the same. For this reason a more general name for this method is chosen; the Black Substrate Method.

THE BLACK SILICON METHOD (BSM) MULTI-STEP ONE-RUN

To solve the problems connected with releasing the etched microstructures, a new technique has been developed which has the ability to etch, passivate, and release MEMS in one run. This technique, the so-called BSM multi-step one-run process, is developed on an Electrotech, Plasmafab 310-340 twin deposition/etch parallel-plate system operating at

13.56 MHz, but is not restricted to that system.

The technique starts with commercially available SOI (Silicon On Insulator) wafers. After the deposition of a 30 nm (lift-off) mask for the pattern definition, the movable structures can be fabricated in only one RIE run with four individual steps): 1) The (an)isotropic RIE ($SF_6/O_2/CHF_3$) of the top Si, 2) the RIE (CHF_3) of the insulator together with the passivation (C_xF_y film) of the sidewalls of the structures, 3) the RIE ($SF_6/O_2/CHF_3$) of the floor, and 4) the RIE (SF_6) of the bulk Si. The process can be finished with a conformal step coverage of a C_xF_y film to protect the released structures from the environment [14]. For instance, these fluorocarbon (FC) films do have an extremely low surface tension and therefore they repel water and others. With this technique it is possible to release long thin Si beams successfully. Examples of the steps are summarised below:

Process step	Description	Etch/deposit rate
1. Photolithography	Shipley 1805	500 nm
2. Metal deposition	E-beam: Cr 25 nm	6 nm/min
3. Lift-off	Acetone	
4. Trench etching	$SF_6/O_2/CHF_3$	0.5-1 μm/min
5. Wall passivation	CHF_3	20 nm/min
Oxide etching	CHF_3	50 nm/min
6. Floor etching	$SF_6/O_2/CHF_3$	1 min
7. Releasing	SF_6	0.5-1 μm/min
8. FC deposition	CHF_3	20 nm/min

In practice, xy-stages, micro grippers, springs etc. with typical dimensions listed in table A were produced and compared with the SCREAM (Single Crystal Reactive Etching And Metallisation) [9] and SIMPLE (SIlicon Micromachining by single step PLasma Etching) [16] processes. As can be seen, the only limiting step is the aspect ratio of trenches and beams.

Table A

Typical dimensions of BSM one-run process

Beam structure	SCREAM	SIMPLE	Invention
Height (μm)	< 20	< 4	< 400
Width (μm)	< 5	< 4	< 50
Length (μm)	< 2000	< 2000	< 2001
Lateral gap (μm)	> 1	> 3	> 1
Aspect ratio beam	< 10	< 10	< 50
Aspect ratio trench	< 7	< 7	< 10

There are some important remarks to be considered in the BSM technique i.e.: 1) Anisotropic etching: The profile has to be vertical with a little underetch making it possible to deposit a FC layer where no ion bombardment occurs i.e. under the "roof" of the mask. The profile can be adjusted by using the BSM method. Also RIE-lag can be suppressed by applying this method. When the intermediate insulator of the SOI wafer is reached, the etch process has to be stopped, to avoid unwanted under etching. This is a crucial step because when the SiO_2 is reached, the loading is decreasing causing a strong enhancement in lateral etching. The etching process is stopped by e.g. visual inspection. Typical parameters during etching are $SF_6/O_2/CHF_3$ gas flow = 30/10/7 sccm, power flux = 0.3 W/cm^2, self-bias = 40 V, pressure = 75 mTorr, 3 inch Si loading, and target temperature=10 °C. 2) Sidewall passivation: The deposition of FC is a function of e.g. pressure and self-bias. This layer is protection the sidewalls during isotropic etching. We observed a satisfying coverage of the sidewalls directly under the mask roofs at pressure=20 mTorr, power flux=0.3 W/cm^2, self-bias=600 V, and CHF3 gas flow = 10 sccm. A typical deposition rate for this process is 20 nm/min. Simultaneously, during this step the insulator is etched at a speed of ca. 50 nm/min. 3) Isotropic etching: Before starting the isotropic etching with an SF_6 plasma it is necessary to "clear" the floor of the trenches first with an oxygen-based plasma such as $SF_6/O_2/CHF_3$.

Some remarks about BESOI are: When a relative thick intermediate layer (>0.1μm SiO_2) is used, bending of the beam structure may occur due to compressive stress in the oxide layer. Another problem might be differences in stress between the two bonded silicon wafers, which may introduce bending/buckling of beams.

BSM SISI: A disadvantage of the BSM SOI technique is that –after releasing the free hanging structures– deep trenches are found and the under etch rate is limited due to

the relatively high Si loading. To eliminate this problem we constructed a Silicon on Insulator on Silicon on Insulator (SISI) wafer. Now, a Si layer of (1–2 μm) is used as a sacrificial layer surrounded by two insulators. The deepest lying insulator protects the Si during releasing resulting in a smooth/flat bottom and a small loading thus high lateral etch rate (>1μm/min).

BSM SCS: This technique is strongly correlated to the SCREAM process [9]. Differences are that the BSM SCS process is fluorine-based whereas the SCREAM process is chlorine-based and the passivation of the sidewalls is different. SCREAM uses SiO_2 which is deposited with a different apparatus and stress could be a notorious problem. BSM SCS uses "in-situ" deposited fluorocarbon (FC) to protect the sidewalls. FC has a low Youngs modulus of elasticity and therefore it does not suffer from stress effects like bending or buckling.

BSM EPI: This technique is related to the SIMPLE process [16]. However, SIMPLE is chlorine-based whereas BSM EPI is fluorine-based. Advantages of fluorine over chlorine is the much higher under etch rate for fluorine-based plasmas. Moreover, the doping level is not restricted to highly doped arsenic (As) as a doping impurity.

APPLICATIONS AND CONCLUSIONS

Briefly, the present invention is directed to a new and unique process for the fabrication of deep trenches in to a substrate using a non-toxic and non-hazardous, preferably fluorine-based mixture in an inexpensive "standard" reactive ion etcher with excellent profile control, high aspect ratio, high etch rates, good uniformity, high selectivity, low surface-damage and -roughening. The process provides a significant advantage in the manufacture of deep trenches at very low d.c. bias voltages for their use in e.g. submicron transistor trench isolation, MEMS applications (e.g. electrostatic actuators or smart sensors), and the fabrication of cheap silicon or polymer-based moulds. The formation of grass can be used positively in sunlight collectors and anti-reflection coatings for e.g. laser applications.

It can be stated that the BSM multi-step one-run process is favourable for the releasing of MEMS with long thin beams. It includes the Black Silicon Method as an excellent tool for profile control and to suppress RIE-lag. Instead of SiO_2, a thin metal (30 nm Cr) layer is used as a mask, which has an almost infinite selectivity with respect to Si and creates less additional stress problems (bending). The fluorocarbon layer has a low Young's modulus which prevents stress problems in long thin beams (buckling). The

intermediate layer of SOI prevents the beam for hollowing out during the isotropic etch making an exact definition of the structure height possible. After the mask is deposited it is now possible to fabricate very quickly, accurate, and at low cost free-hanging MEMS (e.g. an accelerometer, tuneable spring/filter, AM/FM modulator, or micromechanical transistor) in one process run with a RIE plasma without turning the plasma of.

Wafers which are purposely not cleaned or even oxidised in an oxygen plasma and etched in the Black Silicon Regime can be used as an optical diffuser for e.g. laser applications. It is possible to create spikes at well-defined locations in order to form a tip for the use in AFM applications. In our study we are mainly interested in the use of the Black Silicon Method for MEMS applications. In figure 1, a micromachined xy-stage is shown. The structure is etched during one run with standard RIE. After the directional etching, the sidewalls are passivated using a low pressure CHF3 plasma and the xy stage is etched free with the help of an isotropic SF_6 plasma. In the same run the structure is passivated with a fluorocarbon layer using a high pressure CHF_3 plasma [14].

Although the Black Silicon Method is described for the $SF_6/O_2/CHF_3$ plasma, it will also works for other silicon etch gases e.g. CF_4, NF_3, SiF_4, CF_3Br, CCl_4 or Cl_2. In fact, every plasma mixture which consists of a chemical etchant, a passivator and an ion source can be used for the Black Silicon Method, even when the substrate is not silicon at all but e.g. a polymer. All together it is shown that the Black Silicon Method is a very strong tool for etching high structures with excellent profile control using an $SF_6/O_2/CHF_3$ plasma.

The black silicon method (BSM) is a powerful tool in finding recipes for the fabrication of MEMS building blocks (trenches, needles) such as, scanning probe tips, multi-electrodes for neuroelectronic interfaces, micro filtration systems, shadow masks, suspensions for rigid disk data storage, micromoulds, submicron trenches for IC-applications, gratings for biomedical and optical applications, membrane structures for tunable IR filters, integration of sensors and actuators with Integrated-circuits and components for liquid handling systems (e.g. pumps valves)

The following specific advantages and applications are contemplated:

Using the above-mentioned process, deep trenches can be etched in silicon or polymers using the SF_6/O_2 gas mixture to which CHF_3 or another halocarbon may be added. If desired, the silicon etch process is independent of crystal orientation and doping.

Also, deep trenches can be etched in silicon or polymers with excellent profile control. Isotropic, positively and negatively (i.e. reverse) tapered as well as fully vertical

walls are achieved by controlling the plasma chemistry (i.e the gas flows, the pressure, and the power density).

Furthermore, deep trenches can be etched in silicon or polymers with aspect ratios ranging of at least 10, or in case of polymers, up to 20 or even 30. When using cryogenic cooling and/or new plasma sources, such as Inductively Coupled Plasma (ICP), an aspect ratio of 20 or higher can be obtained in silicon. Etch rates ranging up to 5 microns per minute and an etch uniformity better than 5% over the wafer can be achieved.

Also, deep trenches can be etched in silicon or polymers with mask selectivity greater than 10,000 or even greater than 100,000 for metals and greater than a thousand for silicon dioxide.

The deep trenches can be obtained in silicon or polymers with a surface roughness lower than 100 nm of trenches more than 100 microns deep.

After deep trench etching one may prefer to passivate the sidewalls by means of e.g. the same RIE apparatus with a halocarbon coating using a low pressure CHF_3 plasma or another halocarbon plasma.

Devices can also be passivated completely with a halocarbon polymer using a high pressure CHF_3 or other halocarbon plasma. The deposition can be performed at the target plates (i.e. the powered electrode) of the RIE, in the plasma glow, or downstream. As a result of this variation in ion and/or photon impact, the properties of the deposited coating/polymer can be varied as desired.

Devices can also be made using the formation of grass. Silicon sunlight collectors can be manufactured, wherein the surface etched is completely black as a result of the black silicon regime. The black silicon absorbs all the incoming light, making a high efficiency sunlight collector.

Another use of the black silicon is as an anti-reflection coating in e.g. laser applications.

Using the present method, devices for electro-mechanical transduction can be etched, also called Micro Electro-Mechanical Systems (MEMS). Especially, the high aspect ratios obtainable by the present process allow submicron spaced capacitor plates to be etched. In figure 1 a micromachined xy-stage is shown fabricated with the help of the BSM SCS technique. Figure 2 shows the essential steps of the present process.

Also, deep submicron trench isolation for e.g. vertical transistors can be etched. An important feature of the invention is the very low bias voltage which is needed to create

these openings, and as a result, electronics are not damaged during the trench etching.

The present process also allows the production of moulds for duplication applications. For these applications slightly positively tapered moulds are needed with a low surface roughness. Release of the mould is made much easier by the deposit of an anti-sticking layer on top of the freshly etched silicon mould. After the filling and hardening of the duplication polymer in the silicon mould, the duplication polymer is released easily because of the low adhesion of the anti-sticking layer. The anti-sticking layer can be deposited with e.g. the same RIE apparatus, using the halocarbon plasma described above.

Scanning probe tips, needles: All kinds of tips can be created with profiles and radii on request for AFM, STM, MFM applications. Sharp positively tapered silicon tips for AFM applications can be fabricated with the BSM in allowing a controllable under etching. It is possible to fabricate spikes having an aspect ratio of 50 or more and a tip radius smaller than 5 nm. Changing the chemistry in a different direction (eg. more CHF3) creates negatively tapered profiles. These probes can be used for filter or MFM applications. The same approach can be used for fabrication of an array of needles for 3D neuro-electronic interface devices for neuromuscular control and also needles for injection of DNA into cells can be fabricated.

Microfiltration systems: Micro filtration sieve membrane sieve for industrial and biomedical applications.(e.g inkjet filters for printers, blood filtration, beer filtration))

Shadow masks: For the fabrication of high resolution mask patterning in deep holes and its application to an electrical wafer feed through.

Suspensions for rigid disk storage media: Silicon micromachined slider suspension with integrated friction forces sensors for rigid disk storage media.

Micromoulds: A variety of mould inserts (in polymers, semiconductors, metals, insulators) can be fabricated, either for electroplating and/or for moulding and embossing processes. These mouldings can also be used for direct patterning in polymers. For instance for filter applications

Gratings: Gratings with dimensions of 0.1 micron up to hundreds of microns for bio-medical applications and optical applications can be constructed.

Submicron structures: Submicron trench etching for IC-applications (DRAM, SRAM devices).

The black silicon *one run process* is a powerful tool for the fabrication of movable structures for micro electromechanical systems (MEMS) using single crystalline silicon

(SCS) substrates, polymer substrates, metal substrates (Ti) or multilayer substrates (e.g. SOI, BESOI, SIMOX, epiwafers with buried layers and SISI multilayer wafers).

With these technique all kind of movable structures (devices) can be constructed. For instance electrostatically driven xy-stages for AFM, STM, MFM and XPS applications and stepping motors for high resolution positioning over large distances Other examples are accelerometers with displacement sensor, electrostatic voltameter, static friction sensor, impact test sensor, resonant microstructure, electromechanical filter, vibromotor optical shutter, tuning fork rate gyroscopes, electromechanical transistor, micrograppers, fibercutters, logic elements., micromotors, microturbines, robotics, active joints, microflies, microphones, microrelays, microswitches, and gas flow meters.

<u>Example</u>

Starting wafer: p- or n-type, e.g. 300 microns thick SCS, SOI or EPI.

1) Spin-on resist and pattern it with the mask lay-out of interest.

2) Deposit a thin (e.g. 50 nm) metal layer (e.g. Cr, Al, Ni, or Y) by means of evaporation, sputtering, or other suitable deposition methods.

3) Remove the resist with e.g. acetone in order to remove the metal mask locally (also known as the lift-off technique). Eventually, one may decide to deposit the metal layer first and then the resist, after which the pattern of interest is etched in the metal layer using the patterned resist as a mask.

4) Etch the silicon or polymer by means of reactive ion etching (RIE) using an $SF_6/O_2/CHF_3$ gas mixture with typical gas flows ranging up to 200 sccm SF_6, up to 100 sccm O_2, and up to 100 sccm CHF_3, power densities ranging up to 2 W/cm*cm, and pressures ranging up to 200 mTorr (26.7 Pa). The addition of CHF_3 is not essential for the high aspect ratios and may be omitted.

For many applications already formulated these four steps are sufficient. For releasing structures, however, additional steps are necessary:

5) A fluorocarbon film is deposited to protect the sidewalls during isotropic etching (the releasing step 8) using the same RIE reactor ant without breaking the vacuum, Typical plasma setting: Pressure = 20 mTorr, power reflux 0.3 W/cm^2 and CHF_3 flow = 10 sccm. A layer of approximately 100 nm is deposited in 5 min. Simultaneously, for SOI wafers, the insulator is etched during this step at a speed of about 50 nm/min.

6) Before starting the isotropic etching, it is necessary to clear the floor of the trenches with

a short $SF_6/O_2/CHF_3$ plasma or the like.

7) To release the structures, a pure SF_6 (or other fluorine-based) plasma is started, optionally mixed with nitrogen or oxygen to increase fluorine atom concentration in the plasma and thus the etch rate.

8) Before breaking the vacuum and testing the devices, it is possible to protect the devices from moisture or dust by way of a conformal fluorocarbon layer.

DESCRIPTION OF THE FIGURES

Figure 1 shows a micromachined xy stage etched withe help of the present Black Silicon Method in an $SF_6/O_2/CHF_3$ plasma.

Figure 2 shows the various steps of (a) SCREAM-SCS [ref. 9], (b) SIMPLE-EPI [ref. 17] and (c) the present method (BSM-SOI). Step i: deposition and paterning mask; step ii: anisotropic etching; step iii: deposition PECVD oxide protection; step iv: local reomval PECVD oxide / FC deposition; step v: isotropic etching; step vi: deposition metal contacts.

Figure 3 is diagram showing the influence of power, pressure and flow on the profile.

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CLAIMS

1. A process for producing micromechanical structures using Reactive Ion Etching (RIE), wherein a substrate is etched with a silicon etch gas mixture, characterised in that etching is performed until an aspect ratio of at least 10 is obtained.

2. A process according to claim 1, wherein an aspect ratio of at least 20 is obtained.

3. A process for producing micromechanical structures using Reactive Ion Etching (RIE), wherein a substrate is etched with a silicon etch gas mixture, comprising the steps of:

a) anisotropic etching using a first silicon etch gas to obtain a primary microstructure;

b) depositing a halocarbon film on the walls of the primary microstructure;

d) isotropic etching using a second silicon etch gas, to obtain a final microstructure;

said steps being carried out in a single run.

4. A process according to claim 3, further comprising the step of:

c) etching the floor of the primary microstructure using said first silicon etch gas.

5. A process according to claim 3 or 4, wherein said first silicon etch gas in step a) comprises SF_6, CF_4, NF_3, SiF_4, CF_3Br, CCl_4 or Cl_2, in particular SF_6, and oxygen, optionally combined with a fluorohydrocarbon, in particular CHF_3.

6. A process according to any one of claims 3–5, wherein said halocarbon film is deposited in step b) using a fluorocarbon, in particular CHF_3.

7. A process according to any one of claims 3–6, wherein said second silicon etch gas comprises SF_6.

8. A process according to any one of claims 3–7, further comprising the step of:

e) depositing a halocarbon film on the surface of the final microstructure.

9. A process according to any one of the preceding claims, wherein an ion energy of between 10 and 90 eV, preferably between 10 and 50 eV, in particular between 10 and 20 eV, is applied.

10. A process according to any one of the preceding claims, wherein a pressure between 50 and 200 mTorr (6.7 - 26.7 Pa) is applied.

11. A process according to any one of the preceding claims, wherein said substrate is single crystal silicon (SCS), epitaxially grown silicon wafers (EPI), silicon on insulator on silicon on insulator wafers (SISI), silicon on metal, or especially silicon on insulator wafers (SOI).

12. A process for producing trenches in a substrate using standard Reactive Ion Etching (RIE), wherein, prior to the etching, a test substrate is treated with the silicon etch gas mixture, the process parameters including flows of the etch gases, pressure and ion energy, are adjusted until the treated surface of the test substrate is black, optionally a carbon-halogen compound such as CHF_3 is then added to the etch gas mixture and its flow is adjusted until the treated surface of the test substrate is clear again, and then the adjusted process parameters are used for the subsequent etching.

13. An etched silicon structure having a profile with an aspect ratio of at least 10, the structure being obtainable by a process according to any one of the preceding claims.

14. An etched silicon structure according to claim 13, which is a needle for e.g. STM applications.

15. An etched silicon structure according to claim 13, which is a tip haing a radius of less than 10 nm.

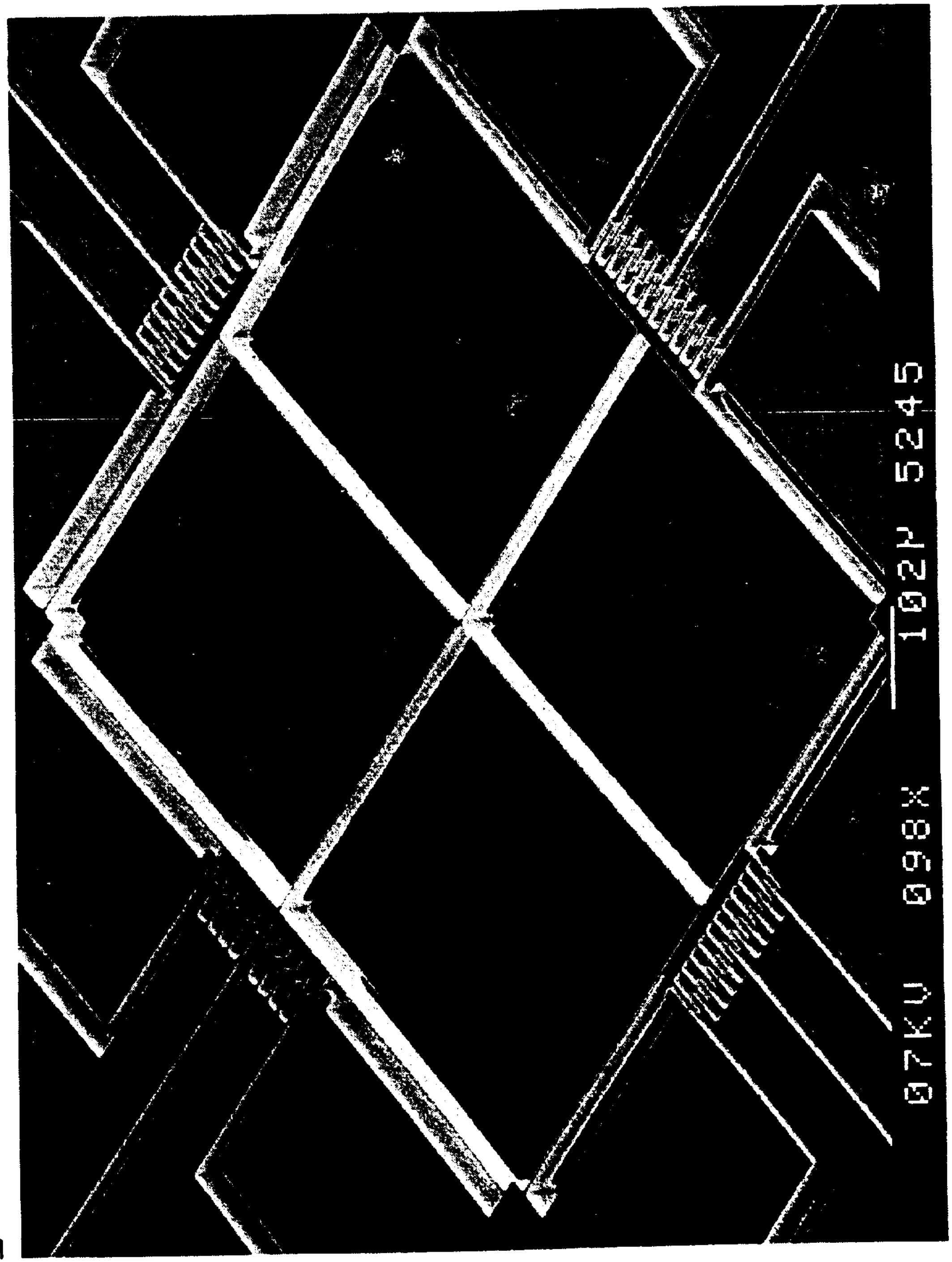


Fig-1

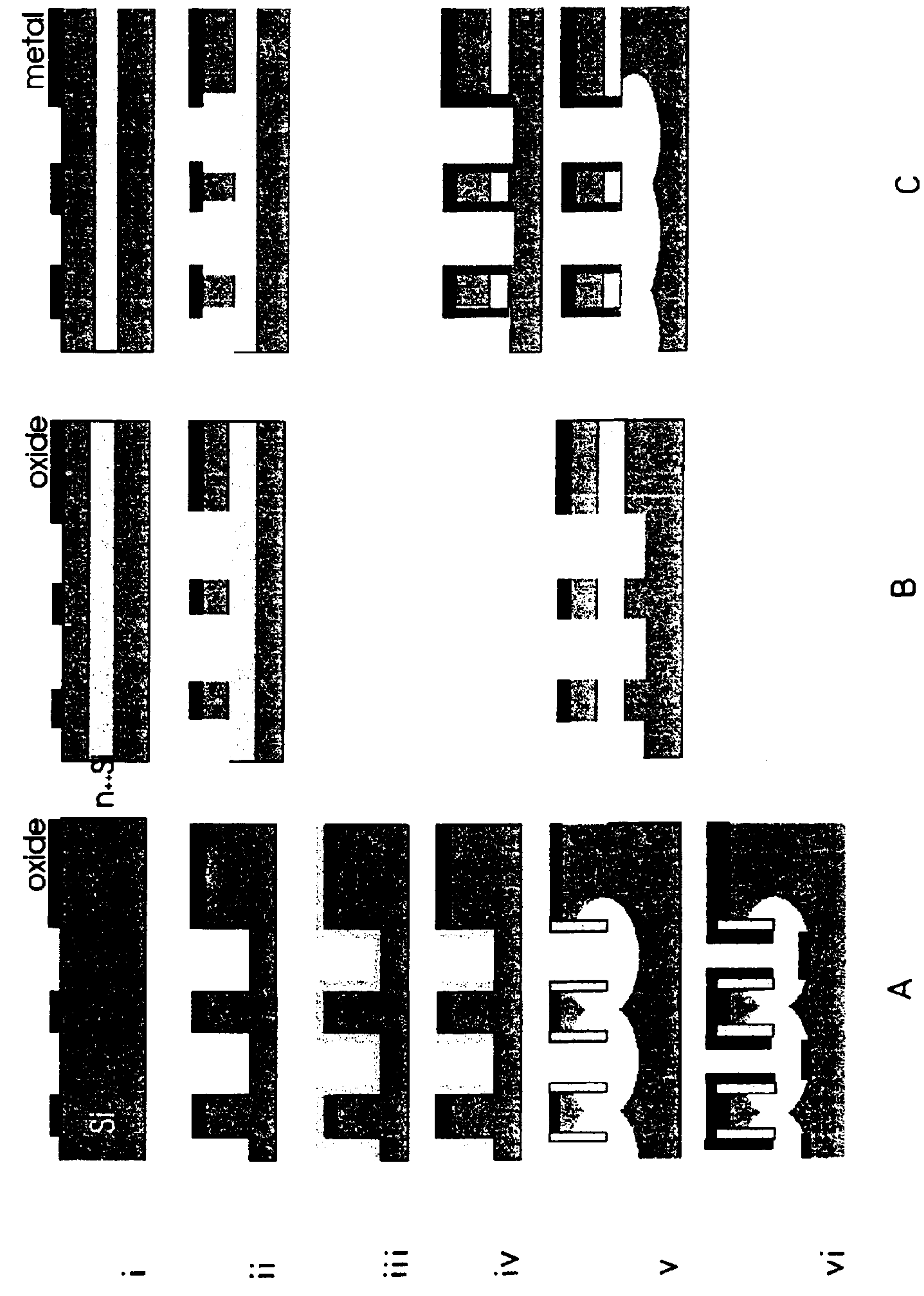


Fig-2

fig-3a

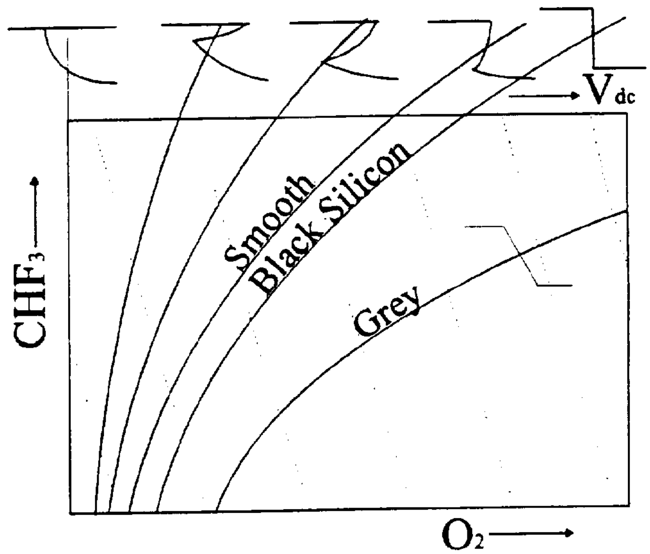
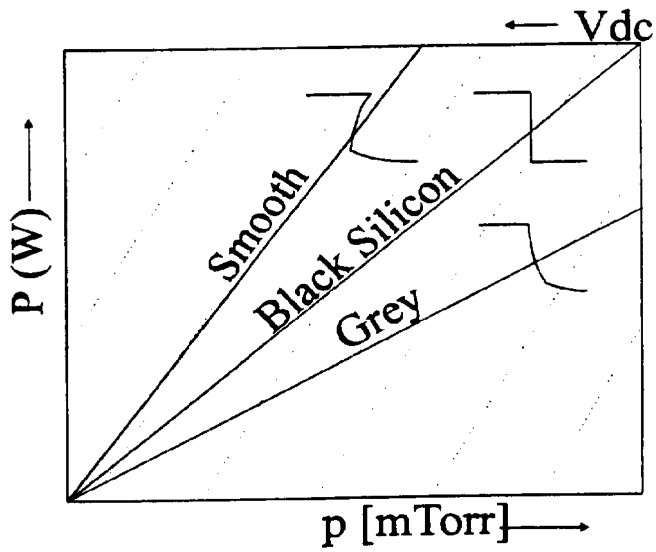

Vdc
Smooth
Black Silicon
Grey
CHF3
O2

fig-3b


Vdc
Smooth
Black Silicon
Grey
P (W)
p [mTorr]

INTERNATIONAL SEARCH REPORT

International Application No
PCT/NL 95/00221

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 H01L21/306 G01N27/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 H01L G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	EP,A,0 413 040 (IBM) 20 February 1991 see column 5, line 6 - column 6, line 35; figures 3A-3D	1,2 12-15
X Y	EP,A,0 565 212 (APPLIED MATERIALS INC) 13 October 1993 see abstract; figures 1-6; table 1	1 13
A	APPLIED PHYSICS LETTERS, vol. 59, no. 14, 30 September 1991 pages 1717-1719, XP 000250007 DAVIS R J 'IMAGE POTENTIALS AND THE DRY ETCHING OF SUBMICRON TRENCHES WITH LOW -ENERGY IONS' see page 1718, right column; figure 3	1,2,9

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search	Date of mailing of the international search report
1 September 1995	18.09.95

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Roussel, A

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	US,A,5 198 390 (MACDONALD NOEL C ET AL) 30 March 1993 see column 3, line 45 - column 5, line 38; figures ---	3-15
Y	EP,A,0 536 968 (NIPPON ELECTRIC CO) 14 April 1993 see column 2, line 41 - column 4, line 6; figure 4 ---	3-13
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Information on patent family members

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